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Title	The Study on the Reaction of Acetylene under High Pressure. (III) : Synthesis of N-Vinyl Phthalimide
Author(s)	Furukawa, Junji; Onishi, Akira
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2, 5-Dimethoxyallylbenzene was isomerized to 2, 5-dimethoxypropenylbenzene by potassium hyroxide and the hyrobromide addition compound was condensed by reduced iron powder to 3, 4-bis-(2, 5-dimethoxyphenyl)-hexane* (yellow oil, b.p. 215–220°/5 mm.) The meso compound* which crystallized (yield 11%) melted at 118–119°. The demethylation gave 3, 4-bis-(2, 5-dihydroxyphenyl)-hexane,*m.p. 219–220°; tetraacetate,*m.p. 149–150°.

α-Diisosafrole was treated with phosphorus pentachloride in refluxing toluene and decomposed with iced aqueous sodium carbonate solution yielding white crystals of carbonyldioxy derivative of diisosafrole,*m.p. 152.5–153°, yield, 37%.

Demethylation of metanethole with methylmagnesium iodide gave 1-ethyl-1-(p-hydroxyphenyl)-2-methyl-6-indanol* in colorless needles, m.p. 158–159°.

The antioxidative property and the toxicity of these compounds together with 3, 4-bis-(m, p-dihydroxyphenyl)-hexane* and 3, 4-bis-(m, p-caraonyldioxyphenyl)-hexane* (Sisido, Nozaki and Kuyama, J. Org. Chem., **14**, 1124 (1949)) were tested in the laboratory of Prof. Takata. The results will be published elsewhere.

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30. The Study on the Reaction of Acetylene under High Pressure. (III)

Synthesis of N-Vinyl Phthalimide

Junji Furukawa and *Akira Onishi*

(Oda Laboratory)

N-vinyl phthalimide is now prepared by pyrolysis of β-acetoxyethylphthalimide (over-all yield 50%) and the polymer is a new source of polyvinylamine. It is claimed in one patent that the compound was also obtained by the reaction of C_2H_2 and phthalimide at high pressure and temperature with Hg salt as catalyst. (C.A. **35**, 3266^7 (1941)). But our repetition of this method disclosed that no vinyl phthalimide could be obtained and metallic Hg was liberated.

Attempts were then made to accomplish this reaction by means of various catalysts, including $(CH_3COO)_2Zn$, Zinc stearate and $(CH_3COO)_2Cd$. $(CH_3COO)_2Cd$ has proved successful. The others were less effective. Phthalimide potassium was also used, but without success. The results are shown in the following table.

Catalyst (g)	Reactn. Temp.	Reactn. time	vinyl P.I.	Resin
Zn Stearate 3	220°C	9 hrs.	0.5g (10%)	5.3g
$(CH_3COO)_2Cd$ 5	200	2	3.0g (50%)	6.0
$HgCl_2$ 6	190	3	0	2.0

P.I. = phthalimide. P.I. 20g and xylene 40g are used in all cases. Yields are calculated allowing for recovered P.I.

One of the experiments was carried out as follows. A 100ml-autoclave was charged with 20g phthalimide, 40g xylene and 5g $(CH_3COO)_2Cd$ and C_2H_2 was pressed in to 12kg/cm.^{2} After shaking for 2 hrs. at about 200°, the pressure fell from 57 to 50kg/cm^2. The reaction mixture was filtered from catalyst and unchanged phthalimide, and was washed with xylene. N-vinyl phthalimide was obtained on the steam-distillation of the filtrate at slightly reduced pressure; yield 3.0g (50%). It melts at 85–86° and is polymerized with benzoylperoxide. A mixed melting point with N-vinyl phthalimide, from β-acetoxyethylphthalimide (m.p. 85–86°) showed no depression.

The new method gives as much yield as the pyrolysis method, but may be of great value on account of the cheapness of the reagents. The authors wish to thank Yamada of the Osaka Industrial Research Institute for the sample of N-vinyl phthalimide from β-acetoxyethylphthalimide.

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31. Studies on Application of Ketone Resins. (VIII)

Application of Acetone Formalin Resin as Adhesives. (7)

Yasuaki Kōzai

(Nodzu Laboratory)

This report deals with the experiments of the Acetone Formalin Resin as an adhesive for the wood. Conditions the application were simlar to those described in the previous paper, (R. Nodzu, R. Gotō and Y. Kōzai: "Wood Research" Bulletin of The Wood Research Institute, Kyoto University, 4 (1950) 50; This Bulletin, 23 (1950)) except the method of heating which was carried out by a radio-heater (wave-length 6 m, power 300–500 W).

1) Examples of the results:

Pressure (Kg/cm^2)	Headening reagent	Radio-heating hour (Kg/cm^2)	Adhesive strength (Kg/cm^2)	Wet adhesive strength (Kg/cm^2)
17	NaOH	1–3	120–130	40–45
	$Ca(OH)_2$	2–5	130–150	45–50
0	NaOH	2–3	50–60	15–20
	$Ca(OH)_2$	0.3–0.5	5–10	0

Test piece: BUNA (3×2×1cm)

2) The addition (1–2%) of wood powder to the resin, gives a good effect.
